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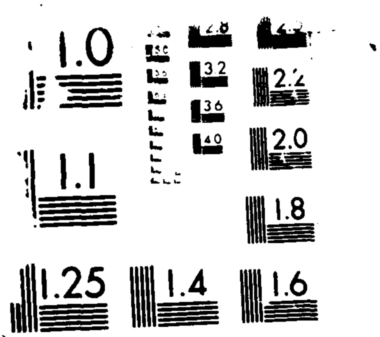
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LITHOGRAPHIC EVALUATION OF COPOLYMERS WITH ENHANCED DRY ETCH RESISTANCE

by

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Electron beam lithographic performance of copolymers of alpha methyl sytrene with maleic anhydride (PMAAMS), and with methyl maleate (PMMAMS) has been evaluated. RIE rates have also been determined with the purpose of developing a resist system with both adequate sensitivity and dry etch resistance. Poly(alpha methyl sytrene) has been shown to exhibit an RIE etch rate of half that of PMMA[1], but its sensitivity is poor due to its low G-scission ($G_{(s)}$) of 0.25 scissions/100 ev[2,3] However, the alternating

copolymer of AMS with maleic anhydride (PMAAMS) exhibits a G_s similar to that of PMMA (G_s = 0.62 - 0.94)[3,4]. These findings were suprising because it had previously been expected that maleic anhydride would crosslink upon irradiation. The present work indicates negligible crosslinking. The G_s of the AMS copolymer with the monomethyl ester of maleic anhydride was previously reported as 1.6[3], but the present work with both gamma and electron irradiation indicates higher G_s values by a factor of almost 2.

Previous sensitometry of PMAAMS was very limited due to low molecular weights and other problems[3,5]. In the present work optimization of molecular weight and work with the methyl maleate copolymer has yielded greatly improved sensitivity. For exposure doses of 40-50 $\mu C/cm^2$, no thinning of unexposed resist was observed using PMMAMS with a 120^{o}C prebake. PMMAMS reverts to PMAAMS at higher prebake temperatures, and thus optimization of prebake conditions has been an important consideration. Preliminary experiments have also been conducted to utilize the reactivity of the anhydride group to improve sensitivity and etch resistance.

The dry etch rate of PMAAMS varied from 0.45 to 0.53 times that of PMMA for molecular weights (Mn) ranging from 10,000 to 50,000. The etchant used was CF_4 with 8% O_2. Thus the etch resistance of alpha methyl styrene has been maintained after copolymerizing with maleic anhydride for sensitivity enhancement.

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Lithographic evaluation of copolymers with enhanced dry etch resistance

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Abstract

Alternating copolymers of alphamethylstyrene (AMS) with maleic anhydride (MA) and methyl maleate (MeM) are evaluated as positive electron resists. The chain scission efficiency (G_s) of P(AMS-MA), determined by exposure to 50 keV electrons, is 0.90 scissions/100 eV. When the maleic anhydride in the copolymer is reacted with sodium methoxide to form its methyl ester, P(AMS-MeM), the G_s increases to 2.9 for electrons and to 3.5 for gamma radiation. Based on these G-scission values, this copolymer is expected to exhibit enhanced sensitivity, while having good dry etch resistance due to the aromatic nature of alphamethylstyrene.

Lithographically, P(AMS-MeM) is more sensitive than P(AMS-MA), as expected from G-scission data. Film properties such as adhesion are also superior for P(AMS-MeM). Using a one hour prebake at 140°C, 10% thinning of unexposed P(AMS-MeM) occurs upon development of pads exposed to an incident electron dose of 8 $\mu C/cm^2$ (accelerating voltage = 20 kV). The contrast (γ) is 2.0 for development of 12 $\mu C/cm^2$ exposures. In comparison, P(AMS-MA) exhibited 10% thinning for an incident dose of 40 $\mu C/cm^2$, which is similar to observations with PMMA. The copolymers are developed with mixtures of ethyl 3-ethoxypropionate and 1-methoxy-2-propanol acetate.

The dry etch rate of P(AMS-MA) in CF_4 plasma with 8% O_2 varies from 45 to 53% of the etch rate of a PMMA standard. The etch rate of P(AMS-MeM) after a 140°C prebake is about 65% that of PMMA. Thus, much of the etch resistance of alphamethylstyrene is maintained in copolymers with maleic anhydride or methyl maleate, while the copolymer with methyl maleate also exhibits significantly enhanced sensitivity.

Introduction

Polyalphamethylstyrene (PAMS) has been shown to dry etch (RIE) at half the rate of polymethylmethacrylate (PMMA)[1], but its sensitivity is poor due to its low chain scission efficiency (G_s = 0.25 scissions/100 eV)[2,3]. However, an alternating copolymer of AMS with maleic anhydride, P(AMS-MA), exhibits a G_s similar to that of PMMA (G_s = 0.62-0.94 scissions/100 eV)[3,4]. These results were suprising because it had previously been expected that maleic anhydride would crosslink upon irradiation due to the presence of alpha hydrogens on the main chain (Fig. 1). It is hoped that this copolymer would exhibit good etch resistance because of the aromatic structure of the AMS repeat unit.

Despite these promising indications, previous sensitometry of P(AMS-MA) has been very limited due to low molecular weight and other problems[3,6]. In the present work, we have optimized the conditions for lithography with P(AMS-MA), but more importantly we have evaluated the methyl ester of MA in a copolymer with AMS. Preliminary work with this methyl maleate copolymer, P(AMS-MeM), indicated a G-scission of about twice that of PMMA[3]. This copolymer therefore promises greater sensitivity than P(AMS-MA) and the potential of maintaining good etch resistance. Previous work with other copolymer systems, such as itaconate copolymers, has demonstrated that forming the mono-methyl ester of an anhydride or di-acid improves some resist properties[5].

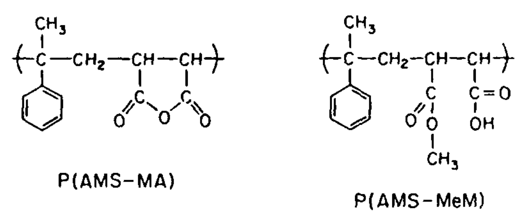

Figure 1. Structures of the alternating copolymers of alphamethylstyrene (AMS) with maleic anhydride (MA) and methyl maleate (MeM).

In the present work, chain scission efficiencies are determined for P(AMS-MA) and P(AMS-MeM) using both electron and gamma irradiation. Lithography with these resists is optimized with respect to prebake conditions, developer and polymer molecular weight. Dry etch rates are determined and compared to a PMMA standard. The intention of this work is to provide a resist with both greater sensitivity than PMMA and improved dry etch resistance.

Experimental

Synthesis and characterization

An alternating copolymer of alphamethylstyrene (AMS) and maleic anhydride (MA) was synthesized in solution at 55°C. Equimolar quantities of the monomers were dissolved in MIBK, and benzoyl peroxide was added at a concentration of 0.08 mmole/L to initiate the polymerization. The reaction was continued for five days. The product, which precipitated as the reaction progressed, was dissolved in acetone and reprecipitated with cold methanol. This polymerization resulted in yields of about 60%.

P(AMS-MeM) was synthesized from the P(AMS-MA) copolymer by reaction with sodium methoxide. A 4.4 g quantity of P(AMS-MA) was dissolved in 100 mL of THF, and 30 mL of sodium methoxide solution (0.022 moles Na in methanol) was added. In this case, sodium methoxide was used only for convenience, but the ester could also be prepared via methanol reflux without introducing possible sodium contamination in the product.

Molecular weights of the copolymers were determined by gel permeation chromatography (GPC) of solutions in THF, using a polystyrene standard. P(AMS-MA) exhibited a number average molecular weight (Mn) of 120,000 and a weight average molecular weight (Mw) of 285,000. After reaction with sodium methoxide the molecular weight was similar, with Mn = 117,000 and Mw = 251,000.

IR spectral analysis was used to confirm the conversion of anhydride to ester after reaction with sodium methoxide and the conversion back to anhydride during prebaking. Silicon wafers were spin coated with the polymers and then prebaked at various temperatures. IR spectra were then obtained by mounting the coated wafer in the specimen beam and an uncoated wafer in the reference beam. The maleic anhydride copolymer exhibited major peaks at 1860 and 1785 cm^{-1}, while the methyl maleate copolymer absorbed at 1740, 1200 and 1170 cm^{-1}, with a very slight peak at 1785 cm^{-1}.

Electron chain scission efficiencies were determined by coating 3-inch silicon wafers with copolymer and exposing the films to a broad beam of 50 keV electrons from a modified RCA transmission electron microscope[7]. Following exposure of a series of films over a range of doses, the molecular weights were determined by GPC. G-scission values were obtained from the slope of plots of 1/Mn versus dose[7].

G-scission for gamma radiation was determined by exposing bulk polymer samples (in nitrogen) to gamma radiation from a ^{60}Co source. A PMMA standard was irradiated simultaneously as a dose indicator using a previously determined G-scission value for PMMA of 0.85 scissions/100 eV[7].

Lithography and dry etching

For lithographic evaluation, films of the polymers were cast onto 3-inch silicon wafers from solutions in 2-methoxyethanol or cyclohexanone. The films were prebaked *in vacuo* for one hour at 120, 140 or 160°C. Patterns were exposed with a Cambridge EBMF-II-150 pattern generator using an accelerating voltage of 20 kV. For sensitometry, a series of 20 µm wide pads were exposed over ten-fold dose ranges. For resolution analysis, patterns were exposed with feature sizes varying from 10 to 0.25 µm. One micron lines and spaces were also exposed. Proximity correction was not used for these exposures.

Patterns were developed with solvents chosen from three-dimensional solubility plots[4]. The optimum developing systems were mixtures of ethyl 3-ethoxypropionate (Kodak EEP) and 1-methoxy-2-propanol acetate (Arcosolve PM acetate). All developing was conducted in stagnant solutions and stopped by blowing with dry nitrogen. A 15-minute postbake at 90°C followed each development. Film thicknesses were measured with a Leitz interferometer and a Tencor profilometer. Resolution was evaluated using a Cambridge SEM.

For determination of reactive ion etch rates, polymer films were prepared as described above and etched in a modified Plasma Therm reactive ion etcher. The etchant gas consisted of 92 vol.% CF_4 at a flow rate of 32.2 SCCM and 8 vol.% O_2 at a flow rate of 2.8 SCCM. The chamber pressure was 30 mTorr, and the power level was 0.2 W/cm^2. Etch rates were determined by measuring film thicknesses before and after a 2.0 minute etch.

The polymethylmethacrylate (PMMA) standard used throughout the experiments was obtained from KTI Chemicals in a 6% solution with chlorobenzene. The nominal weight average molecular weight is reported at 950,000.

Results

The electron chain scission efficiency (G_s) of the alternating copolymer of alphamethylstyrene with methyl maleate, P(AMS-MeM), is 2.9 scissions/100 eV. Using the same technique, a G_s value of 0.9 was obtained for PMMA. P(AMS-MeM) exhibited a G_s value of 3.5 using gamma radiation. These values are nearly twice the previously determined value of 1.6[3]. In any case, these data indicate that the copolymer does not crosslink, either with electron or gamma irradiation, as G_s-G_x is zero. Although the G_s values are only two to three times that of PMMA, the copolymers were investigated further as resist materials because improved etch resistance was expected due to the aromatic alphamethylstyrene constituent.

During prebaking of P(AMS-MeM) films, the methyl maleate unit converts to maleic anhydride. The degree of conversion to anhydride increases steadily with increasing prebake temperature, as determined by IR analysis (Figure 2). Thus, with higher prebake temperatures, some of the film properties and lithographic results are expected to tend towards those of the maleic anhydride copolymer, P(AMS-MA).

The thinning of P(AMS-MeM) copolymers increases with increasing prebake temperature, tending toward that of P(AMS-MA) with higher prebake temperatures (Figure 3). Similar thinning behavior is observed for P(AMS-MeM) when using a 120 or a 140°C prebake, with 10% thinning occurring at a dose of about 8 μC/cm^2 (accelerating voltage = 20 kV). The thinning characteristics of P(AMS-MeM) when using a 160°C prebake are similar to that observed with a PMMA standard, with 10% thinning being observed at 40 μC/cm^2. The developing solvents used were mixtures of ethyl 3-ethoxypropionate and 1-methyl-2-propanol acetate, which are low toxicity substitutes for 2-ethoxyethanol derivatives.

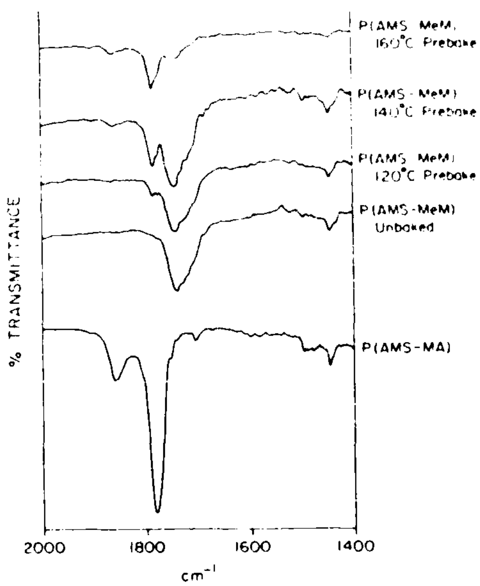

Figure 2. Infrared spectra of prebaked P(AMS-MeM) and P(AMS-MA).

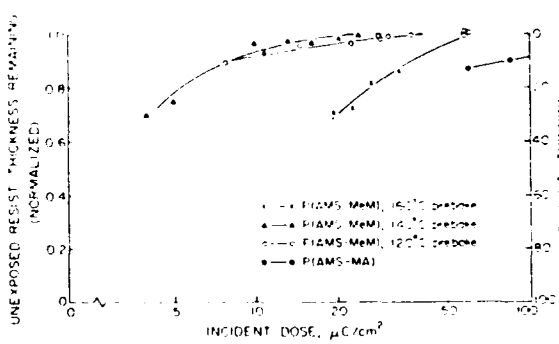

Figure 3. Comparison of thinning curves of P(AMS-MeM) and P(AMS-MA).

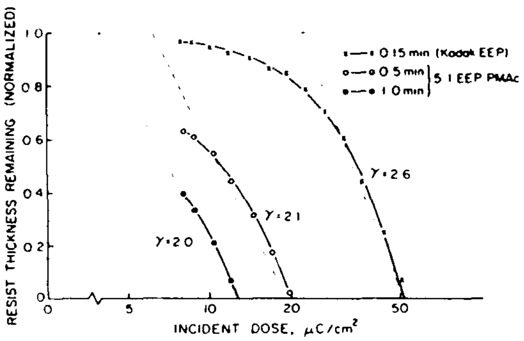

Figure 4. Lithographic response of P(AMS-MeM) with a 140°C prebake.

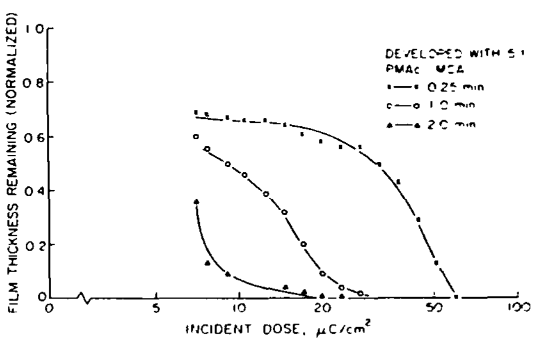

Figure 5. Lithographic response of P(AMS-MeM) with a 120°C prebake, developed with 5:1 1-methoxy-2-propanol acetate (PMAc): 2-methoxyethyl acetate (MCA).

Although thinning is similar for prebake temperatures of 120 or 140°C, slightly superior contrast is observed when using a one hour prebake at 140°C. Lithographic response curves show that reasonable contrast (γ = 2.0 to 2.6) is observed using this prebake condition (Figure 4).

Development of P(AMS-MeM) copolymers was frequently hindered by solubility limits of the copolymer in the chosen developing solvents. Apparently, as the exposure dose is lowered, the molecular weight of the copolymer is not sufficiently reduced to permit thermodynamic solubility in the given solvent. Attempts to develop patterns exposed at doses lower than these solubility limits often resulted in reduced contrast without cleaning out of any lower doses (Figure 5). This problem was alleviated by increasing the strength of the developing system, but this often resulted in reduced contrast and resolution.

SEM photomicrographs of patterns exposed in P(AMS-MeM) show reasonable resolution of one and 3/4 micron features exposed at a dose of 25 μC/cm² using an accelerating voltage of 20 kV (Figure 6). Acceptable formation of one micron lines and spaces is demonstrated in Figure 7.

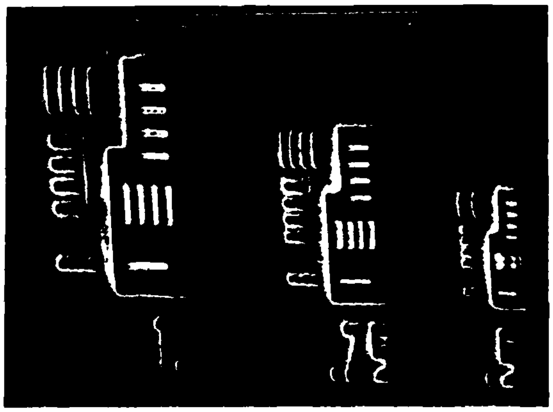

Figure 6. SEM photomicrograph of 1.0, 0.75 and 0.5 μm features in P(AMS-MeM) with a 140°C prebake. This pattern was exposed with a dose of 25 μC/cm² and an accelerating voltage of 20 kV.

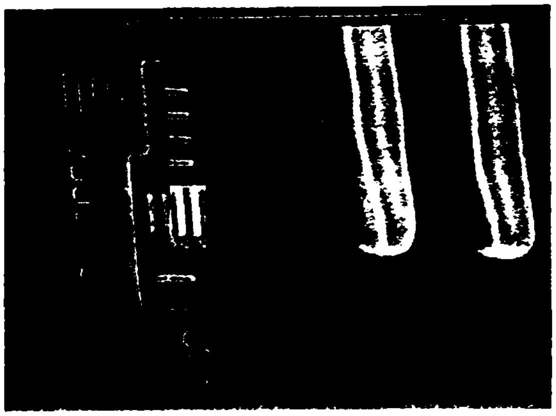

Figure 7. SEM photomicrograph of 1.0 μm features in P(AMS-MeM), exposed with the same conditions as in Fig. 6. Detail at right, showing one micron lines and spaces, is magnified 8x relative to the micrograph on the left.

Table I. Dry Etch Rates of P(AMS-MA) and P(AMS-MeM) Compared to PMMA

Polymer	Etch rate (Å/min).	Etch rate/ PMMA etch rate
P(AMS-MeM), unbaked	1790	0.74
P(AMS-MeM), 120°C prebake	1750	0.72
P(AMS-MeM), 140°C prebake	1550	0.64
P(AMS-MeM), 160°C prebake	1560	0.65
P(AMS-MA)	1420	0.59
PMMA standard	2415	1.00

Dry etch rates in CF_4/O_2 plasma of films of P(AMS-MA) and P(AMS-MeM) were 59 and 74% of the etch rate of PMMA, respectively (Table I). Since the maleic anhydride copolymer is more etch resistant than its methyl ester, the etch rate decreases with increasing prebake temperature as the ester reacts to form the anhydride. Thus, there is a trade-off of etch resistance for sensitivity with decreasing prebake temperature (Figure 4, Table I). However, with a prebake of 140°C, the sensitivity is at a maximum, and the etch resistance is similar to that of P(AMS-MA).

A further versatility of the maleic anhydride copolymers is the ease with which they may be derivatized due to the reactivity of the anhydride. For example, metal-containing compounds could be reacted with the anhydride to further increase the etch resistance of the copolymers.

In conclusion, P(AMS-MeM) exhibits both enhanced sensitivity and enhanced etch resistance, relative to a PMMA standard. The optimum prebake is one hour at 140°C. With these prebake conditions, 10% thinning occurs at a dose of 8 $\mu C/cm^2$, and the etch rate is about 65% of that of PMMA.

Acknowledgements

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